



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>6</sup> :</b> <b>C08J 3/12</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 99/18148</b> <b>(43) International Publication Date:</b> 15 April 1999 (15.04.99)
<b>(21) International Application Number:</b> PCT/US98/21299 <b>(22) International Filing Date:</b> 8 October 1998 (08.10.98)  <b>(30) Priority Data:</b> 08/947,311                      8 October 1997 (08.10.97)                      US  <b>(71) Applicant:</b> H.B. FULLER LICENSING & FINANCING, INC. [US/US]; 1200 Willow Lake Boulevard, St. Paul, MN 55110-5101 (US).  <b>(72) Inventor:</b> FOX, Steve, A.; 1720 Tate Boulevard S.E., Hickory, NC 28602-4246 (US).  <b>(74) Agents:</b> QUAN, Nancy, N.; 1200 Willow Lake Boulevard, St. Paul, MN 55110-5101 (US) et al.		<b>(81) Designated States:</b> AU, BR, CA, CN, JP, KR, MX, NZ, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the</i> <i>claims and to be republished in the event of the receipt of</i> <i>amendments.</i>
<b>(54) Title:</b> COMPOSITION OF POWDER-COATED TACKY MATERIAL AND METHOD OF MAKING SAME  <b>(57) Abstract</b>  A non-tacky material composition comprising a tacky material in particulate form coated with a composite of binder (thermoplastic binder) and powder (i.e., polymer). The resultant particles of the composition have a continuous coating, and thus, are non-tacky, and also, they exhibit free-flowing characteristics and less blocking between particles for a longer time than compositions with a discontinuous coating of powdered polymer on a tacky material absent of any binder.		

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## Description

### COMPOSITION OF POWDER-COATED TACKY MATERIAL AND METHOD OF MAKING SAME

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## Technical Field

This invention relates, in general, to a tacky material composition in particulate form and containing one or more powdered materials, as well as a method to manufacture such compositions. More particularly, the invention relates to tacky material particles coated with binder and powder to render the tacky particles non-tacky.

## Background Art

It has long been commercial practice to microencapsulate pressure sensitive adhesive inside of a shell made of gelatin and/or gum to form adhesive beads that help obviate handling problems due to the tackiness of the adhesive. However, procedures for microencapsulation, such as coacervation, not only are costly, but also require special equipment.

Another approach to help obviate the handling problem from the tacky surface of the pressure sensitive adhesive is to form an aqueous suspension of adhesive cores and then add a polymer to the suspension in order to provide  
5 a bead that consists of a discontinuous polymer coating disposed about the adhesive cores. The cores may be conveniently formed by freezing a pressure sensitive adhesive and then grinding the frozen resultant. Such beads made of a pressure sensitive adhesive core having a  
10 discontinuous polymer coating are disclosed in U.S. Patent No. 5,322,731 to Callahan et al., assignors to Minnesota Mining and Manufacturing Company.

Another approach to help obviate the handling problem from the tacky surface of pressure sensitive adhesives is  
15 disclosed in U.S. Patent Nos. 4,774,138 (Continuation) and 4,576,835 (Parent), both to Gardenier et al., assignors to Henkel Kommanditgesellschaft auf Aktien. These two patents describe tacky adhesive pellets, granules, cubes, flakes and the like that are coated with an anti-adhesive  
20 surface finishing consisting of a solution of (a) a dihydroxy, 3 to 7 carbon, aliphatic alcohol or (b) an aliphatic dicarboxylic or tricarboxylic acid or a water-soluble salt of the acid.

Also, of interest is U.S. Patent No. 4,359,492 to Schlademan, assignor to Atlantic Richfield Company, which shows helping to obviate the handling problem, due to pressure sensitive adhesives normally being tacky, by  
5     dusting adhesive pellets with a friable polymer resin having a ring and ball softening point of at least 95°C.

Of background interest is U.S. Patent No. 4,080,347 to Hefele, assignor to Kufner Textilwerke, which shows a method to modify pressure sensitive adhesives by adding a  
10     melting point and fusion viscosity depressing agent in powdered form to powdered polyamide and tempering the mixture above 55°C to diffuse the added agent uniformly into the polyamide.

General background on the problem of tackiness during  
15     handling of hot melt pressure sensitive adhesives can be seen in the following patents, which show various ways to package a large, solid bulk of adhesive. For instance, U.S. Patent Nos. 5,160,686 and 5,201,420, both to Thaler et al., assignors to National Starch and Chemical  
20     Investment Holding Corporation, show depositing a thin coating of castor oil in a cavity, followed by casting a hot melt adhesive into the cavity. U.S. Patent No. 5,373,682 to Hatfield et al., assignors to National Starch

and Chemical Investment Holding Corporation, shows packaging a hot melt pressure sensitive adhesive mass by directly pouring molten adhesive into a cylindrical plastic tube that is in contact with a heat sink. U.S. Patent No. 5,387,623 to Ryan et al., assignors to H.B. Fuller Company, shows a method of packaging a hot melt pressure sensitive adhesive by solidifying a mass of the adhesive, such as with cold water, and placing the solid in a package made from a film of biodegradable polymer.

U.S. Patent No. 5,401,455 to Hatfield et al., assignors to National Starch and Chemical Investment Holding Corporation, shows packaging hot melt pressure sensitive adhesives by lining a rigid mold with plastic film and then pouring molten hot melt adhesive into the lined mold and allowing the adhesive to solidify.

Accordingly, there has been a long-felt need for a simple method and resultant particulate product of a tacky material, such as a hot melt pressure sensitive adhesive, that does not have a tacky exterior surface, and that obviates the prior art problems from parts of the surface still being tacky and subject to blocking because of a polymer coating being discontinuous on the particles and

from packaging of the tacky material as a large, unitary bulk mass rather than as small discrete particles.

#### Summary and Objects of the Invention

Therefore, the present invention provides a non-tacky material composition comprising tacky particulate material having a continuous composite coating wherein the continuous composite coating includes (a) a coating of thermoplastic binder suitable for extrusion directly adhered onto the surfaces of the tacky material particles and (b) a coating of powder incorporated into the coating of binder.

Additionally, the present invention provides a method for uniformly distributing a continuous coating onto tacky material to form a non-tacky material composition in particulate form. The method comprises first contacting a particulate tacky material with a thermoplastic binder to form a coating of the binder directly adhered onto the tacky material particles. Then, the method comprises contacting the binder-coated tacky material particles with a powder to form a composite coating of the powder and binder directly adhered onto the surfaces of the tacky material particles, and achieving a composition of resultant non-tacky particles that comprise the tacky

material particles having thereon a continuous composite coating that includes the binder and the powder. Preferably, the method is begun with the tacky material cold so that it is temporarily de-tackified.

5           Accordingly, it is an object of the present invention to provide a non-tacky particulate composition of tacky material particles coated with a composite coating of binder and powder, where the composition is in a form that is ready for shipping, and also, where the composition  
10 does not re-block (like the prior art particles of adhesive and discontinuous polymer coating) during long periods of storage prior to use.

          It is an advantage of the present invention, that since the resultant particles coated with binder and  
15 powder are already in particulate form, they are in a form ready for extrusion, mixing, and/or vacuum conveying by conventional means.

          It is a feature of the present invention that the resultant particles coated with binder and powder are  
20 typically dry and non-tacky, and thus, typically exhibit dust-free and free-flowing behavior.

          Some of the objects, advantages, and features of the invention having been stated, others will become evident



as the description proceeds, in connection with the Laboratory Examples described below.

Detailed Disclosure of the Invention

5 The non-tacky polymeric composition of the present invention includes tacky material particles, such as beads of hot melt pressure sensitive adhesive, and the like, coated with a thermoplastic binder and powder. The resultant yields a non-tacky surface on the tacky material particles.

10 More specifically, tacky material particles are first coated with the binder (usually in liquid form), for instance by first tumbling the two ingredients in a drum, and second, a coating of powder is applied, such as by sprinkling dry powder onto the coated mixture (which is  
15 wet if the binder was in liquid form) in the drum, while still tumbling. (Large drums that slowly rotate are commercially available, and include an inlet for air, if desired, to be forced into the drum.) Upon addition of the powder, the resultant mixture quickly (usually within  
20 a few seconds) becomes discrete particles coated with the binder and powder composite. The resultant mixture, even

if still damp, exhibits no tendency for the coated particles to stick together.

The resultant is then removed from the drum, and, if the binder and/or powder is with a solvent (such as in aqueous form with water) and is thus wet, as further described below, the resultant is allowed to dry, typically by spreading it out on a screen and leaving it to dry for about 0.25 to 3 hours so that the solvent evaporates. Longer drying times may be employed with large amounts (i.e., hundreds of kilograms) of tacky material, binder, and powder. The drying can simply be air drying at standard room temperature of about 72°F (22.2°C), but can be at ambient temperatures, including with heat, such as at about 80°F (26.7°C) to about 100°F (37.8°C), or even higher. Higher temperatures will lessen the drying time. Alternatively, the drying may be achieved by continuing to tumble the particles by rotating the drum, optionally with forcing air into the drum to achieve quicker drying.

As noted, the contacting with binder and then with powder is advantageously accomplished in a drum, with rotating to help create uniform distribution of the binder and the powder. The rotation should be for a time

sufficient to achieve uniform coating with the powder incorporated into the binder, and typically the drum is rotated from about 2 seconds to about 20 minutes, more typically about 0.1 to about 10 minutes, and even more typically about 0.5 to about 5 minutes, per coating. Of course, on a factory scale with large amounts (i.e., hundreds of kilograms) of tacky material, binder, and powder, the rotating may be longer (1/4 to 1/2 hour, or even longer) to help achieve uniform coating. A suitable time can be readily determined by the person of ordinary skill in the art without undue experimentation.

The resultant non-tacky composition of tacky material coated with adhesive binder and powder is suitable for extrusion by well known extrusion processes using conventional extrusion equipment. Advantageously, the resultant is free-flowing, and thus, may be readily supplied to a conventional hopper feeder (which moves polymeric resin pellets by vacuum) for extrusion, mixing, et cetera. In the case where the tacky material employed is a hot melt adhesive, the resultant coated particles, when placed for mixing in a hot melting tank for their end use, will melt quickly, yielding a great throughput.

The powder employed in the present invention may be polymeric or non-polymeric.

The term "polymer" or "polymeric" as used herein generally includes, but is not limited to, homopolymers, 5 copolymers, such as, for example block, graft, random and alternating copolymers, terpolymers, et cetera, and blends and modifications thereof. Furthermore, the term "polymer" shall include all possible structures of the material. These structures include, but are not limited 10 to, isotactic, syndiotactic, and random symmetries.

Non-polymeric materials, such as a particulate clay, talc, silica, metal silicate, or metal salt of an organic acid (such as calcium stearate), may be used as the powder for the coating. Use of such materials for the powder 15 depends on whether the intended end use of the non-tacky resultant composition will tolerate such materials, which typically do not melt under certain typical heat conditions of certain end uses.

A suitable talc is sold under the trade name 9600 20 Series by Polar Minerals of Mt. Vernon, Indiana. Talc 9603 has a particle size of 3 to 4.5 microns; talc 9607 has a particle size of 6 to 8 microns; and talc 9610 has a particle size of 8 to 12 microns. As talc is opaque,

and not miscible, it should not be used as the powder where the opacity will be undesirable in the end use of the continuously coated material. For instance, if the end use is an additive for nylon parts, it is preferred  
5 that the powder is polyethylene or polyamide, which are miscible.

Various polymers (in particulate form), including those noted in the above paragraph, also are suitable for use in the present invention as the powder for the  
10 coating. Examples of such polymers, include, but are not limited to, polystyrene (abbreviated as PS), ethylene vinyl acetate copolymer (abbreviated as EVA), polytetrafluoroethylene-co-hexafluoropropylene (abbreviated as FEP), polyvinyl chloride (abbreviated as  
15 PVC), polyamide (also known as nylon), polypropylene (abbreviated as PP), polyethylene (abbreviated as PE), polyesters, polyacrylics, and combinations thereof. Desirably, the chosen polymer is compatible with hot melt adhesives and other tacky materials, as further described  
20 below.

As is well known to those of ordinary skill in the art, the selected PE may be high density polyethylene, medium density polyethylene, low density polyethylene, as

well as the newer type of structures referred to in the art as linear low density polyethylene and/or linear very low density polyethylene. (Linear very low density polyethylene is also called linear ultra low density polyethylene.) A suitable micronized polyethylene powder  
5 may be purchased under the trade name C-5 from Allied Signal and has a nominal particle size of 5 microns.

A suitable size range for the powdery particles is from about 0.5 micron to about 20 microns, more preferably  
10 from about 1 micron to about 10 microns, and most preferably from about 3 microns to about 5 microns. The desired particle size of the powder may be easily achieved by grinding extrusion grade polymeric resin pellets, which generally come about 3 mm in diameter, or grinding non-  
15 polymeric materials, such as talc or clay. Choosing which non-polymeric material or which polymer to employ can be advantageously determined by the person of ordinary skill in the art without undue experimentation, by considering what is the desired end use of the tacky material  
20 particles continuously coated with binder and powder.

The binder is a thermoplastic material that is extrudable, for instance when used in extruders commercially employed for extruding polymeric resin

pellets into an end product of a flexible plastic film. The thermoplastic binder will not cause problems in the extruder (i.e., screw slippage or smoking) and will not cause problems in the end plastic product (i.e., pin holes when the end product is a plastic film).

The binder may be in the form of a solution, emulsion, or dispersion in a solvent, such as water, alcohol, or hydrocarbons. Binders in the form of dry powders may be employed by admixing with water and/or other solvents. Suitable solutions, emulsions (such as a wax emulsion), or dispersions are about 1 to about 65% by weight solids, more preferably about 30 to about 50 % by weight solids, with the remainder being solvent.

Examples of binders useful in the present invention include, but are not limited to, a vinyl acetate homopolymer, a polyurethane, a polyacrylate, an alkyd, a polyvinyl alcohol, a salt of an acid polymer, a polyurea, a polycarbonate, a polysilicate, and combinations thereof. Polyvinyl alcohol and salts of acid polymers (such as salts of polyacrylic acids), if employed as the binder, should be employed in aqueous form.

A suitable binder is the polyvinyl acetate/ethylene vinyl acetate emulsion available from The Reynolds Company

under the trade name REYNCO 8633, which is a water based wax emulsion. The Reynolds Company also sells an aqueous emulsion under the trade name REYNCO 6933.

A suitable vinyl acetate homopolymer for the binder is available as an emulsion under the trade name ROVACE 661 from Rohm and Haas Company of Philadelphia, Pennsylvania. ROVACE 661 is about 55% by weight of vinyl acetate homopolymer in water, has a pH of about 4.5 to 5.0, and a viscosity of about 600 to 1500 centipoise at 20 rpm and 25°C on a Brookfield RV spindle #3.

A suitable polyurethane for the binder is available as a dispersion under the registered trademark SPENSOL L52-MPW-30 from Reichhold Chemicals, Inc. of Research Triangle Park, North Carolina. SPENSOL L52-MPW-30 is an elastomeric dispersion of about 30% solids polyurethane in a blend of water and N-methylpyrrolidone and amine solvents, has a pH of about 7.5 to 9, and has a viscosity of about 0.3 to 1 stoke at 25°C.

Suitable polyacrylics for the binder are available as aqueous dispersions of rosin ester resins under the registered trademark DORESCO ACW8-6 from Dock Resins Corporation of Linden, New Jersey, and as polyacrylic



based water emulsions under the trade name RHOPLEX WL from Rohm and Haas Company of Philadelphia, Pennsylvania.

The ratio of the tacky material particles, the binder, and the powder should fall within certain ranges to yield a free-flowing, non-tacky composition. There should be a sufficient amount of the binder to cover the surface area of the tacky material particles with a thin film of the binder. The binder-covered particles are then treated with sufficient powder readily to convert the mixture to non-tacky, free-flowing, continuously coated particles.

More particularly, it is preferred that the amount of binder be about 30 to 50, more preferably about 40, parts by weight per about 100 parts by weight of the powder employed. In other words, if about 10 parts by weight of powder as compared to about 100 parts by weight tacky material is employed, then about 4 parts by weight of the binder should be employed, whereby the resultant final product of tacky material, binder, and powder contains about 2% by weight of the binder. It is noted that at least about 1 part by weight of powder and about 0.4 parts by weight of binder per about 100 parts by weight of tacky material particles should be employed to achieve

continuously coated particles having a sufficient amount of powder so that the resultant is effective in its desired end use.

It has been found that for a single pass coating of powder onto tacky material particles already having binder, up to about 10 parts by weight of the powder per about 100 parts by weight of tacky material can be employed without the powder dusting off from the resultant.

Suitable ratios of binder and powder can be readily ascertained by the person of ordinary skill in the art without undue experimentation by simply stirring a small amount, perhaps one third cup, of tacky material pellets in a paper cup with, for instance, sufficient water-based binder to yield a thin continuous film over the surface of all the pellets. While still stirring the wetted pellets, a portion of powder of known weight is slowly added over 5 to 10 seconds until the pellets become coated and are damp but free-flowing. The remaining powder may be weighed to determine the amount of dry powder utilized. Suitable ratios are quite broad and acceptable resultant non-tacky material can be produced over a moderate range of combinations. When desired, the thickness of the

composition can be varied considerably by using larger or smaller ratios of binder and powder to the tacky material pellets.

Also, the process of adding the binder and then the powder can be repeated so that tacky material particles already coated can be coated again with binder and powder up to about 20 parts by weight, about 40 parts by weight, about 60 parts by weight, or even higher of powder to about 100 parts by weight of tacky material particles. Accordingly, different powders can be added incrementally to the same tacky material particles. For instance, tacky material particles can first be coated with polyethylene powder as the powder, and then coated with talc as the powder, if that is suitable for the desired end use.

The smaller the tacky material particles are, then the higher will be the surface to volume ratio of the particles. For instance, if the particle is assumed to be spherical, and the coating of powder is considered to be uniform, then the volumetric ratio of coating to particle is approximately 3 times the coating thickness divided by the radius of the sphere for thin coatings. Therefore, if the particle radius is 0.06 inch (0.15 cm), then for a coating thickness of 0.002 inch (0.005 cm), a 10%

volumetric ratio of powder to particle will be achieved. On the other hand, if the particle radius is 0.03 inch (0.08 cm), the same 0.002 inch (0.005 cm) thickness of powder coating will yield a 20% by volume of powder to particle. Consequently, higher loadings of powder can be achieved with smaller particles for a given coating thickness.

In the method for contacting the tacky material particles with binder and then contacting the binder-coated particles with powder, there is no need for adding heat or pressure during formation of the composition. However, heat or pressure may be employed to alter adhesive characteristics. Moreover, the method may be carried out in the ambient atmosphere since there is no need, for instance, for a blanket of nitrogen gas. Preferably, as illustrated in Example 3, the method is carried out in the cold at about 40°F (4.4°C). Usually temperatures below 50°F (10°C) are suitable.

The tacky materials may include, but are not limited to, a pressure sensitive adhesive (abbreviated as PSA) such as thermoplastic polymeric elastomer (abbreviated TPE) or such as amorphous poly-alpha olefin (abbreviated as APAO), a tacky polymer such as a polymer of ethylene-

propylene diene monomer (abbreviated as EPDM), a rubber, and combinations thereof.

A suitable PSA is a TPE sold under the trade name HM 51-128 by The Reynolds Company, or is an APAO sold under the trade name HM-51-320M by The Reynolds Company. The rubber may be a natural rubber, or may be a synthetic rubber such as butadiene-styrene copolymer or acrylonitrile-butadiene-styrene terpolymer. It is noted that butadiene-styrene copolymer and acrylonitrile-butadiene-styrene terpolymer are sold under the registered trademark SANTOPRENE® by Advanced Elastomer Systems.

Preparation of particles of tacky material, for use in the present invention, is accomplished by well known methods. For instance, when tacky materials are pelletized, such as from either a mixing vessel or an extruder, they exit the pelletizer as tacky pellets. One known way to reduce the tackiness momentarily is to employ cold water, such as at 40 to 50°F (4.4 to 10°C), after the pelletizer and before the pellets proceed to a dryer. Thus, when the pellets exit the dryer cold, they will be temporarily non-tacky, and can be further processed for adding the continuous coating of binder and powder in accordance with the present invention.

Additionally, when the tacky material is in particular a hot melt pressure sensitive adhesive, particles of such adhesives may be prepared with cold water as noted above, or instead, by well known aqueous suspension polymerization techniques, as discussed in the above-noted U.S. Patent No. 5,322,731 to Callahan et al.

Whether the tacky material is a pressure sensitive adhesive, or some other material as discussed above, an appropriate particle size for the tacky material particles to be employed for coating with binder and powder in accordance with the present invention ranges from about 0.5 mm to about 15 mm, more preferably from about 2 mm to about 12 mm, and most preferably from about 4 mm to about 8 mm. Also, a generally spherical shape is preferred.

The resultant particles, having a continuous composite coating of (1) binder and (2) powder on the tacky material, are non-tacky and exhibit dust-free and free-flowing behavior. These behaviors are exhibited even if the resultant particles are still damp from having employed a binder in liquid form with a solvent.

The free-flowing behavior of the resultant remains for a long time of weeks, or even months, which allows for a long storage time at ambient conditions of the resultant

without the problem of re-blocking during storage prior to when the end user is ready to use the resultant. The free-flowing behavior may be subjectively observed visually. Alternatively, the free-flowing behavior may be  
5 determined objectively by employing the test defined in ASTM D-1895-69 by measuring the time for funnel discharge of the resultant non-tacky particulate tacky material composition.

Representative Laboratory Examples are set forth  
10 below for a better understanding and appreciation of the present invention.

#### Laboratory Examples

For Examples 1, 2, and 3 below, 7 mm average diameter essentially spherical pellets of elastomeric pressure  
15 sensitive adhesive (the tacky material) were made using well known techniques as described above using extrusion with cold water between the pelletizer and the dryer. The pellets were then stored in a cold water bath at 40°F (4.4°C) in order to maintain them temporarily detackified  
20 until they were employed as the tacky material. The particular pressure sensitive adhesive employed was a TPE,

which is sold under the trade name HM 51-128 by The Reynolds Company of Greenville, South Carolina.

Example 1 (Comparison)

5 A 5 gram sample of pellets of pressure sensitive adhesive (HM 51-128) was removed from the cold water bath, dried off, and coated with 1% by weight of binder (REYNCO 8633 water based wax emulsion from The Reynolds Company) by stirring for 1 minute in a 9 ounce (300 ml) cup while the pellets were still cold. As the temperature  
10 increased, tackiness of the binder-coated pellets increased. When they reached room temperature, they stuck together and were not free-flowing. This indicated that employing binder only does not prevent pellet-to-pellet blocking of tacky material pellets.

15 Example 2 (Comparison)

A 50 gram sample of tacky material pellets of pressure sensitive adhesive (HM 51-128) was removed from the cold water bath, dried off, and placed in a 9 ounce (300 ml) cup with 2% by weight of powder (C-5 micronized  
20 polyethylene from Allied Signal). The mixture was stirred for 2 minutes, and then, spread out on a screen and



allowed to air dry for 4 hours. Initially, the coated pellets gave the appearance of being non-tacky, but they were very dusty.

5       Next, the coated pellets were allowed to sit at ambient conditions for 2 days. The powder-coated pellets were then poured out of the cup, and were observed to begin to exhibit pellet-to-pellet blocking. This indicated that a coating of only powdered polymer (without any binder) on tacky material pellets is a discontinuous  
10       coating that will, upon storage of the powder-coated particles for a short time of only 2 to 3 days, lose the free-flowing behavior and begin to exhibit pellet-to-pellet blocking behavior.

### Example 3

15       A 50 gram sample of tacky material pellets of pressure sensitive adhesive (HM 51-128) was removed from the cold water bath, dried off, and placed in a 9 ounce (300 ml) cup, together with 1% by weight of binder (REYNCO 6933 aqueous emulsion from The Reynolds Company) and  
20       stirred for 1 minute to achieve coating. Then, 2% by weight of powder (C-5 micronized polyethylene from Allied Signal) was added with stirring to the coated pellets, and

stirring was continued for 2 minutes until the pellets were continuously coated with a composite of binder and powder.

The mixture was then spread out on a screen and  
5 allowed to air dry for 4 hours. Optionally, drying may be achieved in a rotating drum together with passing air into the drum in order to accelerate drying. The coated pellets were observed to be dust-free and free-flowing.

After 3 days at ambient conditions, a portion of the  
10 continuously coated pressure sensitive adhesive pellets was poured from the cup and was observed to exhibit free-flowing behavior. Thus, the particles were still non-sticky, and did not exhibit any pellet-to-pellet blocking.

Then, a portion of the continuously coated pressure  
15 sensitive adhesive pellets was melted, and was observed to be translucent and without agglomerates, which indicates miscibility of the polyethylene powder into the pressure sensitive adhesive. Also, upon melting, the material appeared fully to regain its initial tackiness as a  
20 pressure sensitive adhesive.

Example 4

The procedure of Example 3 was repeated, except this time the powder was talc (9603 from Polar Minerals). However, the HM 51-128 (inside of the resultant coated pellets) was still exhibiting its tacky surface, and hence, the coating was not continuous. Also, since HM 51-128 is a hot melt adhesive, the end use of the resultant coated pellets would be in a hot melt molten bath, and it would be expected that the talc, since it is not miscible, would settle out when the pellets are placed in the bath. Thus, talc is not desirable for use as the powder when the tacky material inside is a hot melt adhesive, such as HM 51-128.

Example 5

A 50 gram sample of tacky material pellets of polymer of EPDM, since the pellets were stuck together in clumps as received, had to be broken apart with force and separated on a flat sheet of paper. These pellets were essentially spherical with an average diameter of 3 mm. They were placed in a 9 ounce (300 ml) cup with 1% by weight of binder (Rovace 661, a vinyl acetate homopolymer emulsion from Rohm and Haas), and stirred briefly

(approximately 30 seconds). Then, 2% by weight of talc (9603 from Polar Minerals) was added and the mixture was stirred briskly for 1 minute.

At that point, the continuously coated pellets were not tacky, and after air drying for 1 hour, remained non-tacky. The pellets were placed in a 6 ounce (200 ml) jar and were observed not to stick together after 2 months.

#### Example 6

A 50 gram sample of tacky material pellets of APAO (6 mm average diameter, essentially spherical pellets of hot melt adhesive HM-51-320M from The Reynolds Company) was removed from cold water, dried off, and placed in a 9 ounce (300 ml) cup, and coated with 1% by weight of binder (Reynco 6933 aqueous emulsion from The Reynolds Company) by stirring. Then, 2% by weight of powder (C-5 micronized polyethylene from Allied Signal) was added and that mixture was stirred until the pellets were continuously coated with a composite of binder and powder. That mixture was air dried for 2 hours.

The continuously coated pellets were then determined to be dry and non-sticky and remained non-sticky in a 6 ounce (200 ml) jar until re-melted in an open glass cup 4

days later. When re-melted, no indication of agglomerates was observed.

#### Example 7

This was a continuous in-line process suitable for  
5 production on a factory scale.

A continuous stream of tacky material pellets (temporarily non-tacky due to being chilled to 40°F, i.e., 4.4°C, and supplied cold) of pressure sensitive adhesive (HM 51-128) was flowing from a Gala pelletizer at 100  
10 pounds/hour (45.4 kg/hour) into a rotating first container (i.e., a bucket of approximately 10 liter volume). By continuous metering, binder (REYNCO 6933 aqueous emulsion from The Reynolds Company) was pumped into the first bucket at 1 pound/hour (0.454 kg/hour) with rotation of  
15 the first bucket at 35 RPMs to achieve coating of 1% by weight binder. Then, the binder-coated tacky material was allowed to flow continuously from the first bucket into a second container (i.e., another 10 liter bucket). By continuous metering so that the resultant would have  
20 powder in the amount of 2% by weight, powder (C-5 micronized polyethylene from Allied Signal) was added by an auger feeder with continued rotation of the second

bucket at 35 RPMs until the pellets were continuously coated with a composite of binder and powder.

The mixture was then allowed to flow into a rotating drum dryer open on both ends so that the mixture air dried  
5 for 15 minutes. Dried material was continuously transferred from the dryer to a shipping box. The coated pellets were observed to be dust-free and free-flowing.

After 20 days at ambient conditions, a portion of the continuously coated pressure sensitive adhesive pellets  
10 was poured from the shipping box and was observed to exhibit free-flowing behavior. Thus, the particles were still non-sticky, and did not exhibit any pellet-to-pellet blocking.

Then, a portion of the continuously coated pressure  
15 sensitive adhesive pellets was melted, and was observed to be translucent and without agglomerates, which indicates miscibility of the polyethylene powder into the pressure sensitive adhesive. Also, upon melting, the material appeared fully to regain its initial tackiness as a  
20 pressure sensitive adhesive.

It will be understood that various details of the invention may be changed without departing from the scope of the invention. Furthermore, the foregoing description

and Laboratory Examples are for the purpose of illustration only, and not for the purpose of limitation-- the invention being defined by the claims.

CLAIMS

What is claimed is:

1. A non-tacky material composition comprising a tacky material in particulate form having a continuous coating adhered thereon, wherein the continuous coating includes a composite of (a) a coating of thermoplastic binder suitable for extrusion directly adhered onto the tacky material particles and (b) a coating of powder incorporated into the coating of binder.
2. The composition of claim 1, wherein the powder, the binder, and the tacky material particles are present in an amount of about 1 part by weight to about 40 parts by weight of powder per about 100 parts by weight of tacky material particles and about 30 parts by weight to about 50 parts by weight of binder per about 100 parts by weight of powder.
3. The composition of claim 1, wherein the powder is a polymer selected from the group consisting of polystyrene, ethylene vinyl acetate copolymer, polytetrafluoroethylene-co-hexafluoropropylene, polyvinyl chloride, polyamide, polypropylene, polyethylene, polyesters, polyacrylics, and combinations thereof.



4. The composition of claim 1, wherein the powder is a non-polymeric material selected from the group consisting of a talc, a clay, a silica, a metal silicate, a metal salt of an organic acid, and combinations thereof.

5 5. The composition of claim 1, wherein the binder is selected from the group consisting of a vinyl acetate homopolymer, a polyurethane, a polyacrylate, an alkyd, a salt of an acid polymer, a polyurea, a polycarbonate, a polysilicate, and combinations thereof.

10 6. The composition of claim 1, wherein the binder, during adhering of the coating of the binder, was together with a solvent, wherein the binder and the solvent were in a form selected from the group consisting of a solution, an emulsion, a dispersion, and combinations thereof, and  
15 the composition is dry and substantially free of the solvent that was present.

7. The composition of claim 1, wherein the tacky material is selected from the group consisting of a pressure sensitive adhesive, a tacky polymer, a rubber, and combinations thereof.  
20

8. The composition of claim 7, wherein the pressure sensitive adhesive is selected from the group consisting

of a thermoplastic polymeric elastomer, an amorphous poly-alpha olefin, and combinations thereof.

9. The composition of claim 1, wherein the resultant particles, having a composite coating of powder  
5 and binder that is adhered onto the tacky material particles, are non-tacky and exhibit dust-free and free-flowing behavior.

10. The composition of claim 1, wherein the tacky material is a pressure sensitive adhesive and the powder  
10 is polyethylene.

11. The composition of claim 1, wherein the tacky material is a polymer of ethylene-propylene diene monomer and the powder is talc.

12. The composition of claim 1, wherein the tacky  
15 material has a particle size ranging from about 0.5 mm to about 15 mm and the powder has a particle size ranging from about 0.5 microns to about 20 microns.

13. A method for uniformly distributing a continuous coating onto tacky material in particulate form to form a  
20 non-tacky material composition comprising:

(a) contacting a tacky material in particulate form with a thermoplastic binder suitable for extrusion to form a coating of the

binder directly adhered onto the tacky material particles, followed by

- 5 (b) contacting the binder-coated tacky material particles with powder to form a coating of the powder incorporated into the binder coating of the binder-coated particles, and
- (c) achieving a resultant non-tacky composition of tacky material particles having adhered thereon a continuous coating that includes
- 10 a composite of the binder and the powder.

14. The method of claim 13, comprising repeating steps (a) and (b) at least once.

15 15. The method of claim 13, wherein the binder is together with a solvent in a form selected from the group consisting of a solution, an emulsion, a dispersion, and combinations thereof, and wherein the method includes step

(d) allowing the resultant non-tacky composition of tacky material particles with a continuous coating that includes

a composite of the binder and the powder to dry to

20 substantially remove the solvent.

16. The method of claim 15, comprising repeating steps (a), (b), (c), and (d) at least once.

17. The method of claim 13, wherein during contacting in steps (a) and (b), the powder, the binder, and the tacky material particles are present in an amount of about 1 part by weight to about 40 parts by weight of powder per about 100 parts by weight of tacky material particles and about 30 parts by weight to about 50 parts by weight of binder per about 100 parts by weight of powder.

18. The method of claim 13, wherein the binder is selected from the group consisting of a vinyl acetate homopolymer, a polyurethane, a polyacrylate, an alkyd, a salt of an acid polymer, a polyurea, a polycarbonate, a polysilicate, and combinations thereof.

19. The method of claim 13, wherein the powder is a polymer selected from the group consisting of polystyrene, ethylene vinyl acetate copolymer, polytetrafluoroethylene-co-hexafluoropropylene, polyvinyl chloride, polyamide, polypropylene, polyethylene, polyesters, polyacrylics, and combinations thereof.

20. The method of claim 13, wherein the powder is a non-polymeric material selected from the group consisting of a talc, a clay, a silica, a metal silicate, a metal salt of an organic acid, and combinations thereof.

21. The method of claim 13, wherein the tacky material is selected from the group consisting of a pressure sensitive adhesive, a tacky polymer, a rubber, and combinations thereof.

5        22. The method of claim 21, wherein the pressure sensitive adhesive is selected from the group consisting of a thermoplastic polymeric elastomer, an amorphous poly-alpha olefin, and combinations thereof.

10       23. The method of claim 13, wherein the resultant particles, having a composite coating of powder and binder that is adhered onto the tacky material particles, are non-tacky and exhibit dust-free and free-flowing behavior.

15       24. The method of claim 13, wherein the tacky material is a pressure sensitive adhesive and the powder is polyethylene.

25. The method of claim 13, wherein the tacky material is a polymer of ethylene-propylene diene monomer and the powder is talc.

20       26. The method of claim 13, wherein the tacky material has a particle size ranging from about 0.5 mm to about 15 mm and the powder has a particle size ranging from about 0.5 microns to about 20 microns.

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 98/21299

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 6 C08J3/12

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 960 187 A (H. STOCK ET AL.) 1 June 1976 see claims 1,2,6,7 see figure 5 see column 5, line 65 - column 7, line 33	1,3,7-10
X	WO 97 12695 A (NITECH CORPORATION) 10 April 1997	1-6,9, 12-20, 23,26
Y	see claims 1-19	1-9, 11-23, 25,26

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

\* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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"&" document member of the same patent family

Date of the actual completion of the international search

26 January 1999

Date of mailing of the international search report

09/02/1999

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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A	US 3 528 841 A (C.R. DONALDSON ET AL.) 15 September 1970 see claims 1-5 see column 4, line 4-35 -----	1,3,10

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